Dr. F. Zumstein sen. - Dr. E. Assmann
Dr. R. Koenigsberger - Dipl. - Phys. R. Holzbauer
Dipl. -Ing. F. Klingseisen - Dr. F. Zumstein jun.
Patentanwälte
8 München 2, Bräuhausstraße 4

0021362

D E S C R I P T I O N

of an invention having for a title:
"PROCESS AND DEVICE FOR THE MANUFACTURE OF REINFORCED CONCRETE SLABS."

In the name of : MONTEDISON S.p.A. & MOPLEFAN S.p.A., both Italian Comapnies with head offices in Milan (Italy) - Foro Bonaparte 31.

Named inventors : Andrea VITTONE, Pierpaolo CAMPRINCOLI.

****

The present invention concerns a process and corresponding device for the continuous preparation of slabs, panels and the likes, based on hydraulic or water-settable binders, containing incorporated in them net-shaped structures as a reinforcement.

From Spanish Patent n° 460.292 there are known manufactured articles of cement concrete, also in the form of slabs, containing as a reinforcement layers of oriented, synthetic polymer films, fribrilled and spread open to form sort of a net. Such manufactured articles display a rather high mechanical resistance to bending, pulling, impact, fatigues, to gellification and to permeability to water; wherefore, said articles are considered very interesting for applications in the building field.

The incorporation into the binder of one or more of the net-like structures of the mentioned kind, each formed, in general, by a plurality of superimposed fibrilled polymeric films, hardly leads to a uniform and capillary diffusion of the binder in the net-like structure, there being, above all, the risk of the formation of air bubbles and of portions of the unimpregnated reinforcing structure, especially in a manufacturing process running at industrially competitive output rates.

The process of this invention allows to carry out this preparation in a continuous way, at acceptable industrial production speeds, and without the above mentioned drawbacks, even when using reinforcing structures of a very high number of superimposed layers.

Such a process is characterized in that it comprises at least one full series of the following operations from (a) to (f):

(a) continuous deposition of a layer of a cement mix on a horizontal moving ribbon or band, or a porous surface;

(b) continuous feeding of an open-mesh net-like structure, consisting of one fibrilled film, or of several superimposed fibrilled films, on that layer, in the direction and sense in which moves said porous surface;

(c) deposition of the net-like structure on the surface of said layer;

(d) deposition of a layer of cement concrete (mix) on such a net-like struture;

(e) compacting the whole, consisting of the layers of cement mix and of the net-like structure by means of vibration carried out at least in the vertical sense;

(f) compression of the whole assembly or slab, with the consequential reduction of its content in water [redacted] [redacted] to values comprised between 25% and 35% of the weight of the solids present in it.

The process of this invention may consist of one single series of operations from (a) to (f), or it may comprise any wanted number of such operational series, in succession, one after the other, with the formation of slabs containing any wanted number of net-like structures alternated to layers of cement mix.

By "cement mix" one must intend a mixture entirely consisting or mainly consisting of one or more hydraulic or water-settable binders such as e.g.: cement, chalk, mortar and the likes, with water and, in particular the mixtures

having a volumetric ratio water/binder comprised between 30-50/100.

To the mix there may be added different types of additives for various purposes, such as for instance:

- inert materials such as e.g.: quartzy sand, with the purpose of improving the dimensional stability and duration of the finished manufactured article;
- fluidizing chemical agents, known products that in general act in the way to reduce the water requirements of the cement mix or concrete;
- well known chemical agents acting as hardening, accelerating or retarding agents;
- chemical agents well known as water-repellents which act to reduce the absorption of water by the hardened cement and improve the performance of the finished slab;
- coloured pigments, to be used in connection with white cement, for the production of coloured slabs;
- water-emulsified synthetic resins that polymerize inside the cement structure, thereby improving the resistance and elasticity characteristics of the finished slab, according to already known techniques;

- short surface-reinforcing fibres, such as: asbestos, cellulose fibres and derivatives therefrom, alkalino-resistant glass fibres, short polyolefine fibres, steel fibres, etc., and in general all the products or materials susceptible to be mixed with the cement.

In fact, a characteristic of the process of this invention is that of allowing, by means of a differential dosing or metering of each layer, the differentiating of the type of mix in the various layers, depending on the particular characteristics one wishes to impart to the slab, with a wide range of possibilities of variation and with the possibility to save in production costs.

For instance, it is possible to use high-resistance cement, which is more expensive, only in the two outer layers, or there may be carried out impermeabilizations with water-repellent agents, or reinforcing treatments with either emulsified synthetic resins or with reinforcing fibres, only in above mentioned layers, with an evident saving on the cost of raw materials. Analogously, the production of coloured slabs may be carried out by using white cement and/or pigments only in the outer layers.

Furthermore, inert fillers may be added only to the innermost layers of the laminate in correspondence with the neutral deformation axis, thereby achieving a saving on the cost of cement and of reinforcement.

The distribution of the flows relating to the deposition of the cement mix layers before and after the deposition of each net-like structure, is made so as to ensure both the complete covering up of the net as well as a uniform presence of the nets in the cement of the intermediate layers.

In the case the slab is prepared using nets of a small number of fibrilled films, which therefore are more easily penetrated by the cement, it is sufficient to have only one dosing or metering device for each net position, unless one desires to obtain slabs of alternate layers of net and cement, in which case at the beginning of the first and of the last series of operations from (a) to (f), there must always be a free metering device or doser forming the initial and the last superficial layers.

The net-like structures, used as reinforcement in the process of this invention, may be of any kind of synthetic polymer capable of forming fibrillable films.

Said net-like structures may be obtained starting from synthetic polymer films, by using known fibrillating methods, capable of imparting to the film by a successive cross-wise spreading an open-mesh net-like structure.

A method suited for preparing such structures is de - scribed, for instance, in English Patent n°1.073.741. The net-like structure used in the process of this invention may consist of either just one single fibrilled film or of a plurality of superimposed fibrilled films, possibly welded together.

A method of preparation for composite, net-like structures, consisting of a plurality of superimposed fibrilled films and utilizable in the process of this invention, is described, for instance, in Italian Patent n°22.800 A/79, filed in the name of the same Applicant.

The compacting operation (e) is carried out by means of devices that transmit a vibration, in an at least vertical sense, to the cement mix and that preferably hit the nets bedded in the cement layers contemporaneously downwards and with a high frequency, without, however, creating points of stopping of the advancing net.

The vertical vibratory action may also be combined ._

with horizontal vibrations imparted to the surface of the conveyor belt or porous surface.

The compacting operation causes the penetration of the pasty matrix into the net-like reinforcement, acting under pressure against the porous surface, and at the same time bringing about an intimate and uniform contact between matrix and reinforcement, eliminating air bubbles and zones of non-impregnation. The compacting, in general causes a slight loss of water through the porous surface. This water may be allowed to flow freely downward or be gathered, for instance, by means of suction systems with a very slight depression.

Lastly, the compacting may be carried out in combination with vibrating high-frequency movements, imparted to the conveyor belt itself, in the impregnation zones, by electrical or mechanical vibrators, according to known cement vibration techniques.

The compression operation (f) aims at regulating or adjusting the thickness of the slab, at compressing the cement and to eliminate the air as well as the water in excess with respect to the content, considered necessary, of 25-35% by weight on the global weight of nets and cement, and moreover to smoothen its surface.

Before this operation the slab may, however, be subjected to a preliminary dehydration operation by suction under vacuum of the water. Likewise after the compression operation (f), the slab may be subjected to an additional vacuum suction treatment, with the purpose of further reducing the content in water down to the value necessary to confer to it a consistency sufficient for possible successive processing operations such as, for instance, lateral (side) trimming, cutting, undulation,,etc., before the final hardening sets in.

The device, which forms the further object of this invention, is characterized in that it comprises one or more operational units arranged in series, each of which consists:

(a') a net-like open-mesh structure feeder, the structure consisting of one or more superimposed fibrilled films;

(b') a horizontal porous surface in motion;

(c') a device for guiding the net-like structure over the moving porous surface;

(d') at least one feeding-dosing device for the deposition of the cement mix on said surface or on said net-like structure;

(e') a vibrator, vibrating at least in the vertical sense, to be used for compacting the cement mix;

(f') a compressor for compressing the cement mix.

The feeder (a') may be represented by a standard reel-unwinder, reels on which the net-like open-mesh structures are wound up, and works maintaining the tension of said structure low and constant.

The device (c'), acting as a guide for the net-like structure, may consist of a system of either smooth or fluted or grooved rollers, or it may consist of rotating, free-wheeling or motor-driven bushes, and will serve to approach the net-like structure to the horizontal porous surface and/or to deposit it onto the layer of cement mix deposited on said horizontal porous surface.

The porous surface (b'), acting as conveyor belt, may consist of a heavy fabric, of the type of paper-mill felts or for cement-asbestos mixes.

Such a surface may have either a levelled or profiled cross-section, with any profile it is wished to impart to the slab, and may moreover, be provided with reliefs, gaskets or side blades, in order to ensure the lateral containment of the cement mixes.

That surface moves in the same direction and sense of the net-like structure feeder.

The feeder-doser (d') must be such as to ensure cha racteristics of constancy and uniformity of flow of the cement mix on the porous surface and/or on the net-like structure

This device may may consist of various devices of dif ferent types, such as for instance:

- a horizontal over-flow (surge) tank fitted with an inter nal stirrer, in order to avoid the hardening of the mix,, and suitably fed according to the already known techniques, by a volumetric pump;
- a vat provided with a horizontal dosing screw which ensures both the metering by discharg from one side of the vat as well as the advancement of the mix in the vat itself;
- surge pipeline in which, by means of a pump, there is made to circulate a batch of cement mix that is much greaterthan the metered quantity. The dosing or metering, in such a case, is carried out by a volumetric pump which provides to integrate into the circuit the quantity that had flown out of ~~the~~ a gauged and adjustable slot placed on the surge pipeline, or through a series of spray-nozzles with gauged bore.

In general, the metering device will have to allow the easy emptying and washing of the installation during the stops of the machine, while it will also have to avoid flow variations due to clogging or to crystalline deposits on the part of the cement.

Each dosing or metering system, moreover, must be fed by a volumetric pump which shall allow to vary the flows from layer to layer, and more particularly the flow of the last layer which, if a good superficial finish of the slab and a sure covering of the net-like reinforcement are wished, must be dosed in quantities much greater than that of the other layers.

The compacting vibrator (e') may take various forms, one of which is represented by element (8) (vertically acting vibrator) illustrated in figure II, (A) and (B), consisting of a rotating rollers supporting frame (15), the rollers being driven positively and being provided with reliefs, for instance blades arranged radially to the rollers and having a peripheral speed equal to or slightly greater than the sliding speed of the net/cement assembly on band (7).

The roller-carrying frame is connected elastically with a vibrator (16), for instance an electro-magnetical one, carried by an upper supporting frame.

Moreover, the distance between the edges of the blades and the band (7) is adjustable so that the blades be in contact with or draw in the surface of the slab being formed, so as to be able to hit it and set it into vibration.

The slab under formation receives, thus, mechanical stresses of a high frequency by the revolving blades, without however their causing any stoppage of the movement of the slab itself. The compacting device may be achieved also with rigid vertical elements having at their ends sliding elements such as, for instance, idling rollers, spheres, small plates oriented in the sense of motion of the nets, etc.. Or it may be achieved by rows of rotating elements turning in the sense of the net-like structure, for instance wheels, and prompted by the vibrations, to hit rapidly the surface of the nets and of the cement mix.

The vibratory motion which acts vertically, may be obtained,according to known methods, by means of electro--mechanical or pneumatic apparatuses, while the frequency of the vibrations, depending on the speed of the motion of the nets, on the type of nets and on the matrix and the thicknessof the slab, may vary within a very wide range, that is, from just a few cycles/second to several hundred cycles/second.

Compressor (f') may consist of a pair of rollers of which, for instance, the lower one is driven by a motor, and between which it is possible to carry out a distance adjustment and to pass the composite slab onto the conveyor belt; or it may consist of a doctor blade system with a smoothening and thickness-regulating blade.

In order to avoid the adherence or dragging of the cement and/or of the net by the rollers, these latter may be covered or coated with anti-adhesive sunstances, for instance silicon resins, and they may, moreover, be fitted with scraping blades or with nozzles for air jets, in order to favour the detachment (separation) of the slab.

Before and/or after compressor (f') there may be present, in a succession, suction systems for the removal of the excess of water present in the slab.

In figure I is represented in a side view (A) and in a plan view (B), an operational unit of the device of this invention, with the accessory elements suited for its working. In said figure there will be noted:

A feeding device (1) for the feeding of the net-like structure (2); the net-guide (3) (which may be a roller or a rotating brush) which serves to approach said net-like structure to the porous surface (7) in motion in the sense in which the arrow points; the porous surface supporting the

drilled plate (9); the dosing devices (4), fed by volumetric pump (5) through valves (6); a vibrator (8); a compressor (10) consisting of rollers (19) and (20), of which roller (20) is driven by a motor (13); gaskets (17), placed longitudinally on the porous surface, for retaining the cement mix spread on said surface, and for avoiding losses of the mix following lateral spilling of the mix itself.

In fig. I, (C) represents a doctor-blade compression system, with blade (21) for the scraping and regulation of the thickness of the cement slab.

In fig.II there is represented a type of device according to this invention, consisting of three operational units arranged in series, where, additionally, there are represented: the suction systems (14) placed under the vibrators (8); a further suction system (11); a conveyor belt (18) for gathering the final plate or slab; a washing device (12) for the porous carrying surface; rotating blades (19) for the trimming of the slab and the moving cutting lance (20) for the cutting of the slab into crop-ends.

Still in figure II, (B) represents a schematical view of the production steps for a composite slab consisting of three nets (2) alternated with three layers of cement mixes (21).

Referring to fig. I, the carrying out of the operations in the process of preparation through an operational unit, is the following one:

The net-like structure (2) is unwound from the reel by means of the unwinder (1), guided by the net-guiding system (3) on the porous band (7) moving horizontally, and is placed on the cement layer pre-existing on the said conveyor belt, where it will be covered up with a layer of cement coming from the successive doser (4). The assembly of cement/net/ /cement is then conveyed to the vibrator-compacter (8), then to the compression or lamination system (10) and from there it is conveyed towards the successive operational unit, where it suffers the superimposition of a new net-like structure followed by that of another layer of cement.

Passing from one operational unit to the successive one, in a device consisting of various different operational units arranged in series, like the one represented in fig.II, the single laminated elements are superimposed naturally on one another, they combine with each other and compact themselves forming the final humid slab which comes out of the last pressing position.

In order to obtain a good union or joint of the single elementary layer with each other, it is however necessary that the percentage of residual water in each layer and the state of compacteness be such as to allow the diffused fil fration of the successive overlying layers. Thus, the pressurees in the compressor (10) (and the possible suction in the suction systems (14)), will have gradually growing values, growing from the first to the last operational unit and will have to be adjusted (regulated) in such a way that the residual water in each intermediate layer be comprised, as a general indication, to be verified, for each type of plant and each cement/nets composition, within about 0.25 to 0.35 of the water/solids relationship.

In the last operational unit, the final composite slab, after compacting of the last cement/net layer, is preferably subjected to a vacuum phase with the purpose of removing the water in excess with respect to the final level of water/solid relationship of about 0.20-0.25, wherein the slab displays sufficient consistency for the successive processing stages.

The removal of the excess of water may also be obtained in the course of the last compression or lamination phase in the last operational unit which, having to bring the slab to the finally desided thickness, necessarily cre

ates a considerable superficial pressure and increases the contact between the reinforcement and the matrix, eliminating the water in excess or possible air bubbles.

The process according to this invention permits also to produce slabs with different profiles, undualted etc., either by the known undulation or profiling systems on the trimmed and cut slabs, both by the use of a porous surface already having the desired profile, for instance an undulated profile, and of all the organs of the machine and in particular the net-guides, the compactors, the vacuum boxes, the compre ssion or pressure rollers, as well as all the motion, transmission and belt-cleaning rollers having the same profile of the belt itself, so as to maintain the necessary adherence.

EXAMPLE 1 :

Using a device, consisting of 5 operational units arranged in series, each of the type illustrated in fig. I, there were produced flat slabs of cement reinforced with about 8% by volume of net, obtained from a fibrilled polypropylene film.

The slabs showed a finished size after trimming, of: 100 x 200 cm and 7 mm thickness.

The rough width, before trimming amounted to 106 cm. The net of polypropylene showed a weight of 107 g/cc, had open meshes and was 106 cm wide, being formed of 12 layers of polypropylene films each 75 micron thick, fibrilled by longitudinal slitting and subsequently spread open transversally with a 9-fold expansion of its original width, wherefore it suffered a longitudinal shrinkage or contraction of 0.85.

The layers in the net showed the unfibrilled slanting bands arranged approximately at ± 12° and ±25° with respect to the direction or sense of the slitting or fibrillation.

The preparation of the slabs was carried out by sucessively impregnating with a cement mix 5 nets whose total weight amounted to 535 g/linear meter.

The cement mix was of the following composition:

- Portland 325 cement = 100 parts b.w.
- water = 39 parts b.w.
- superfluidizing agent at 20% concentration= 1 part b.w.

The density of the mix was equal to 1.90 g/cu.cm

The feeding rate of each of the five nets, and the speed of the porous surface, were both 12 mt/minute.

The peripheric (peripheral) speed of the rollers forming the

compressor (10), is greater by about 2% than the speed of the porous surface, so as to maintain the slab always in traction (tension).

The dosing of the mix, by means of the volumetric pumps, is adjusted in each operational unit as follows:

1st operational unit: 23.0 lt/min. of which:

- 13 lt/min. to the dosing device before the deposition of the net;
- 10 lt/min. to the dosing device after the deposition of the net.

2nd, 3rd & 4th unit, respectively : 21.0 lt/min. of which:

- 10.0 lt/min. to each dosing device be fore deposition of the net;
- 11.0 Lt/min. to each dosing device af ter the deposition of the net.

5th op. unit : 24.0 lt/min. of which:

- 10.0 lt/min. to the dosing device be- fore deposition of the net;
- 14.0 lt/min. to the dosing device after deposition of the net.

for a total of: 110 lt/min.

The porous surface (or conveyor belt) consisted of a felt of polypropylene fibres, needle-point treated, with a specific weight of 1500 g/sq.mt.

The elimination of the excess water in each unit was regulated as follows:

- in the first units from 1 to 4 := free discharge,
- in the 5th unit: = 350 mmHg of vacuum applied immediately after the 5th compacting device.

The compression rollers were regulated as follows:

			Pressure between rollers (Kg/sq.cm)	Max. distance between rollers
1st	unit	=	10	2 mm
2nd	unit	=	10	4 mm
3rd	unit	=	15	6 mm
4th	unit	=	20	8 mm
5th	unit	=	35-45	7 mm

The compacting system consisted of a frame carrying 4 (four) rollers, each carrying 32 blades, and having a diameter of 120 mm, said compacting system being operated by an electromagnetic vibrator producing vertically directed vibration,

at a frequency of 3000 cycles/minute.

The slab at the outlet of the last compression position showed a content in water, measured by drying in an oven at 105° ÷ 110°C, of 22.7% on the total weight of solids.

The slabs were trimmed at the edges to 100 cm width and then cut to a length of 200 cm.

After staying of the slabs in water for 28 days and ensuing drying in the air for 48 hours, there were measured the unitary resistances at break under bending stresses, by method UNI 3948, thereby obtaining the following results:

- Resistance, in longitudinal direction = 388 Kg/sq.cm
- Resistance in a transversal direction = 137 Kg/sq.cm

The thickness of the sample slab amounted to 6.9 mm, as the mean value of 10 measurements at different points.

EXAMPLE 2 :

With the same machine of the preceding example, with the same type of polypropylene nets and with the same procedures of combination, there were prepared slabs of the following net size: 100 x 200 cm x 7 mm of thickness, using white cement coloured with mineral pigments:

The cement mix was of the general composition:

- white Portland (cement, type 425:	100 parts b.w.
- water	39 " " "
- superfluidizing agent at 20% concentr.:	1 " " "

The dosing or metering device n°1 and n°10, which give place to the formation of the two outside (external) layers of the finished slab, were fed with the above indicated mix additioned with 3.5% b.w. of red iron oxide, while the intermediate metering devices were fed with the general mix.

The process conditions were the same as those used in example 1.

After staying in water and drying in the air, the slabs which on the surface showed a uniform light red tinge, were measured as to their bending resistance with UNI 3948 method, obtaining the following results:

- longitudinal resistance	=	396	Kg/sq.cm
- transversal resistance	=	153	Kg/sq.cm
- thickness, mean value on 10 measurements	=	7.0	mm

C L A I M S

1) Process for the continuous production of slabs, panels and other like manufactured articles, based on water--settable binders, and containing incorporated in them net-like structures made of synthetic polymers as reinforcements, characterized in that said process comprises at least one full series of the following operations, from (a) to (f), carried out in a continuous way:

(a) deposition of one layer of a cement mix on a band or conveyor belt, or a porous surface in a horizontal motion;

(b) feeding of an open-mesh, net-like structure consisting of a fibrilled film or of a plurality of superimposed fibrilled films, onto said layer, in the direction and sense in which said band or porous surface is moving;

(c) deposition of the net-like structure on the surface of said layer;

(d) deposition of one layer of cement mix on said net-like structure;

(e) compacting of the whole assembly consisting of layers of cement mix and of the net-like structure, by vibrations carried out in an at least vertical sense;

(f) compression of said assembly or whole, with the consequential reduction of its content in water to values comprised between 25% and 35% on the weight of the solids present.

2) Process according to claim 1, characterized in that the compacting operation (e) is carried out with a simultaneous downward percussion of the nets embedded in the cement.

3) Process according to claim 1, in which the compression operation (f) is preceded and/or followed by a dehydration phase of the whole or slab, by sucking off the water under vacuum.

4) Device for regulating the process according to the preceding claims, characterized in that said process comprises one or more operational units, arranged in series,? each one consisting of:

(a') a feeder of net-like structures with open meshes, consisting of one or more superimposed fibrilled films;

(b') a horizontal porous surface in motion;

(c') a device for the guiding of the net-like structure on said prorous surface;

(d') at least one feeding-dosing device for the deposition of the cement mix on said surface or on said net-like structure;

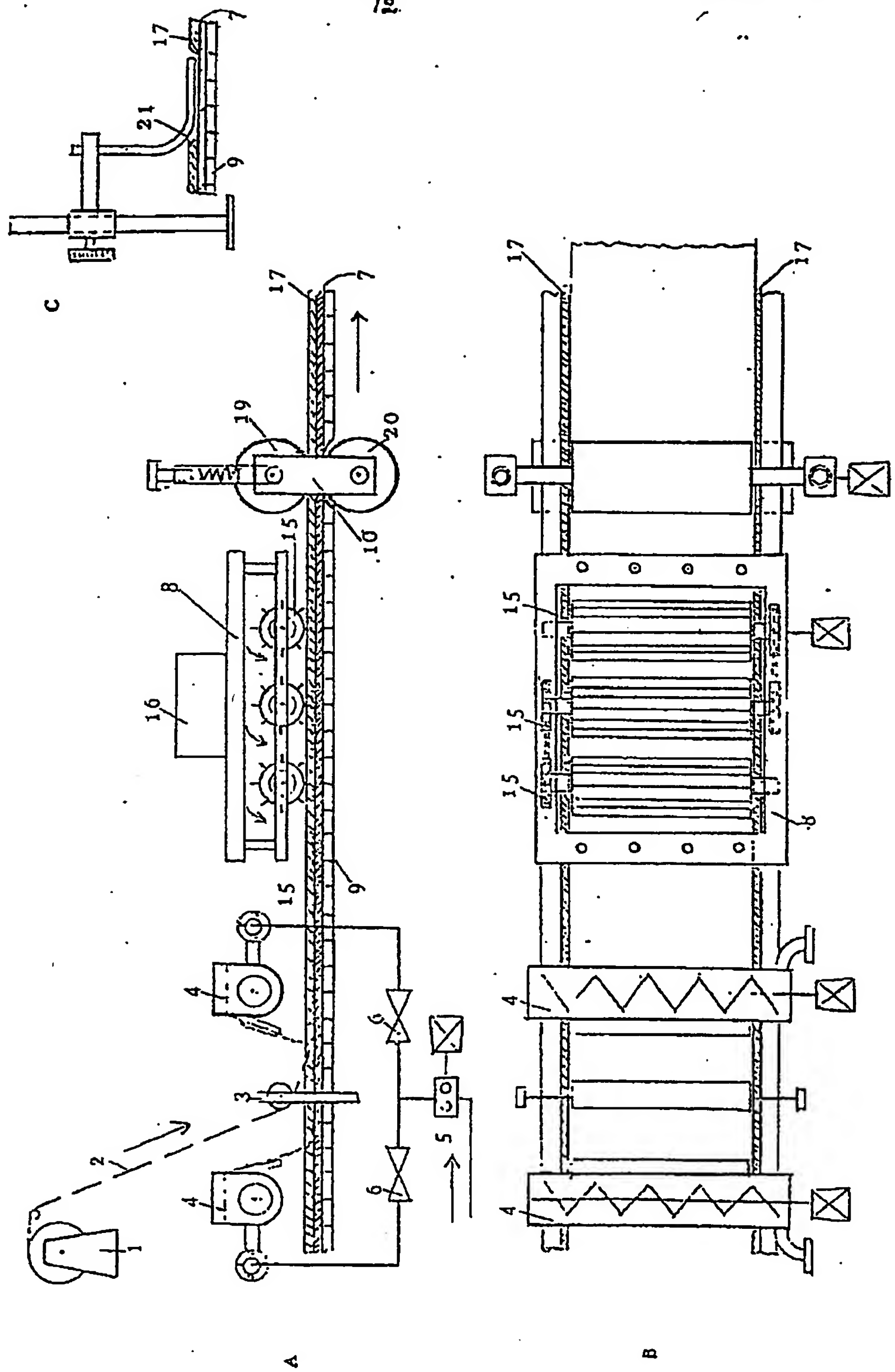
(e') a vibrator, vibrating at least in the vertical sense for the compacting of the cement mix;

(f') a compressor for the compressing of the cement mix.

5) Device according to claim 4, characterized in that it comprises suction devices placed before and/or after the compressor (f').

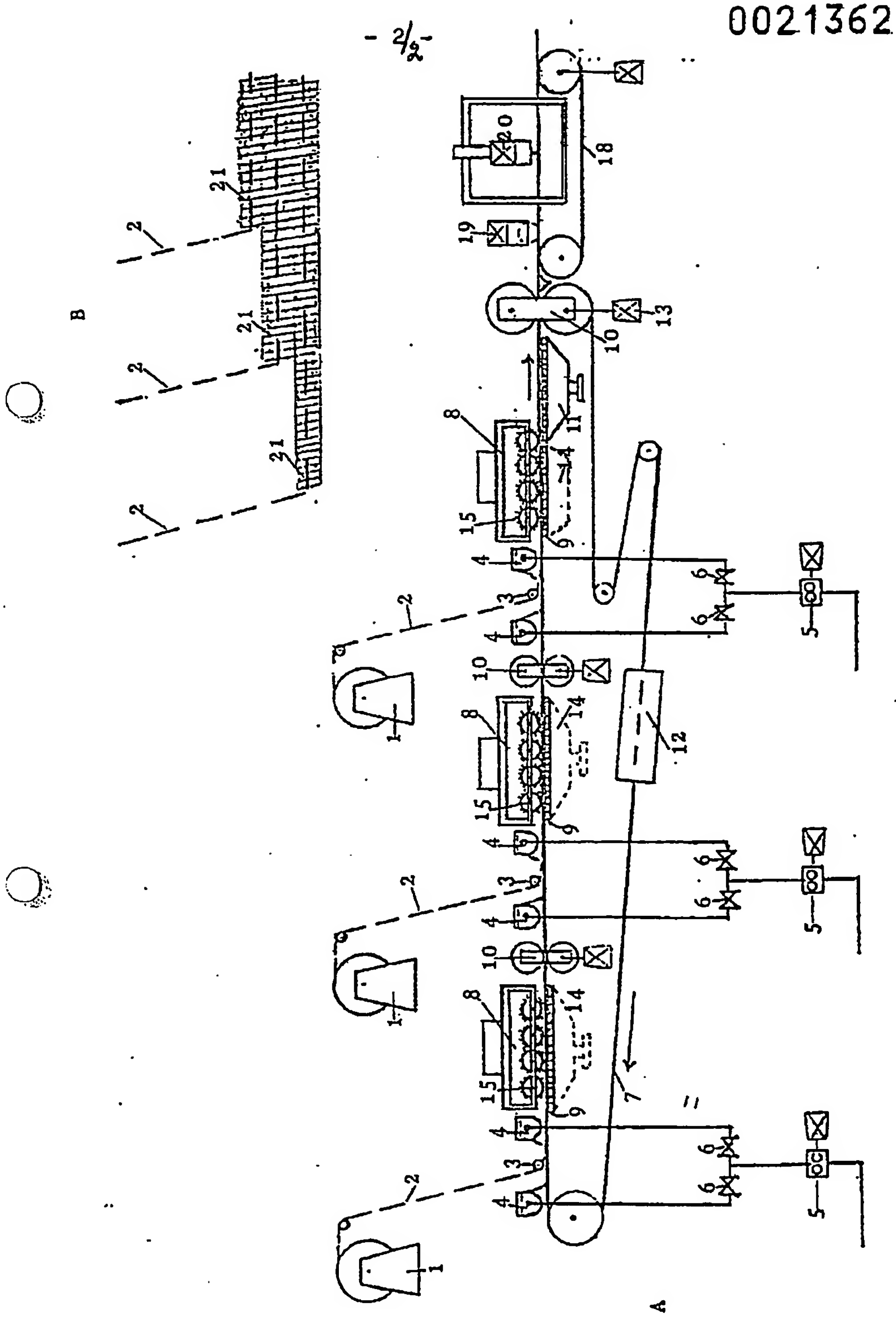
6) Device according to claim 4, characterized in that the vibrator (e') comprises rotating rollers provided with radially arranged reliefs which are in contact with, or draw in the surface of the slab.

FIG. I



0021362

- 2/2 -



0021362

European Patent Office

EUROPEAN SEARCH REPORT

Application number
EP 80 10 3450

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim
E	GB - A - 2 038 701 (STAMICARBON) * Page 4, lines 3-39; figure * & NL - A - 79 08211 & BE - A - 880 149 & FR - A - 2 442 115 & DE - A - 2 946 225	1,3-5
	FR - A - 2 356 610 (UNIVERSITY OF SURREY, HANNANT) * Page 3, lines 28-32; page 5, lines 10-16; page 8, lines 17-24 *	1,3-5
E	EP - A - 0 003 245 (STAMICARBON) * Page 11, line 14 - page 12, line 28; figure 1 *	1,3-5
	FR - A - 923 935 (J.R. DERLON) * Whole document *	1,4
	FR - A - 905 006 (J.C. THOREL) * Page 3, lines 9-40; figure 7 *	1,4
E	GB - A - 2 034 627 (DOW-MAC CONCRETE) * Whole document *	1,3-5

CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)

B 28 B 23/02

TECHNICAL FIELDS SEARCHED (Int.Cl. ³)

B 28 B
C 04 B
E 04 C

CATEGORY OF CITED DOCUMENTS

X: particularly relevant
A: technological background
O: non-written disclosure
P: intermediate document
T: theory or principle underlying the invention
E: conflicting application
D: document cited in the application
L: citation for other reasons

&: member of the same patent family, corresponding document

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	18-09-1980	BOLLEN

EPO Form 1503.1 06.78

(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 803 484 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
29.10.1997 Bulletin 1997/44

(51) Int Cl.6: C04B 28/18, C04B 22/14,
C04B 40/02
// (C04B28/18, 14:42, 18:24,
22:14)

(21) Application number: 97301638.9

(22) Date of filing: 12.03.1997

(84) Designated Contracting States:
BE FR GB

(30) Priority: 22.04.1996 JP 100320/96

(71) Applicant: ASK CORPORATION
Yokohama-shi Kanagawa-ken (JP)

(72) Inventors:
- Masato, Sakiyama
Tsurumi-ku, Yokohama-shi, Kanagawa-ken (JP)
- Takuya, Asami
Yokohama-shi, Kanagawa-ken (JP)
- Tomoki, Iwanaga
Tsurumi-ku, Yokohama-shi, Kanagawa-ken (JP)
- Yasuhide, Oshio
Tsurumi-ku, Yokohama-shi, Kanagawa-ken (JP)
- Masaaki, Oda
Tsurumi-ku, Yokohama-shi, Kanagawa-ken (JP)

(74) Representative: Waldren, Robin Michael
MARKS & CLERK,
57-60 Lincoln's Inn Fields
London WC2A 3LS (GB)

(54) Calcium silicate board and method of manufacture therefor

(57) The object of the present invention is to improve the overall strength and interlaminar strength of a compact (green sheet) before hydrothermal reaction by using alunites, alums and aluminum sulfate having a specified specific surface area which does not require the addition of a setting retarder or a curing accelerator together with a curing agent in order to provide a method of manufacture for a lightweight calcium silicate board which does not give rise to interlaminar peeling or bursting during hydrothermal reaction and to provide such a calcium silicate board. The method of manufacture for a calcium silicate board according to the present invention is characterized by use of one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more.

Printed by Jouve, 75001 PARIS (FR)

Description

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to a calcium silicate board and a method of manufacture therefor, and in particular a lightweight (i.e., bulk specific gravity of 1.0 or less) calcium silicate board and a method of manufacture therefor.

Description of the Prior Art

Conventionally, calcium silicate boards have been widely used as a building material, chiefly for interior trimmings, because they are light, exceptionally easy to work with and dimensionally stable, and they are nonflammable. The molding processes for calcium silicate boards include Hatschek sheet machine process, press molding, and single layer molding, and the calcium silicate boards are manufactured from a compact formed from a raw material slurry, containing calcareous material, siliceous material, and inorganic filler, generally by reacting and curing the compact with saturated water vapor in a pressure vessel.

However, when manufacturing lightweight calcium silicate boards, particularly using the Hatschek sheet machine process, the interlaminar bonding within the compact is weak before hydrothermal reaction and the water content is high. During hydrothermal reaction, this gives rise to thermal expansion of excess water and high vapor pressure, which in turn leads to problems of interlaminar peeling and bursting.

Some of the methods used to overcome these problems are pressing the compact after molding and then subjecting it to hydrothermal reaction, or using a turnbuckle to squeeze the compact during hydrothermal reaction, but employment of these methods increases the bulk specific gravity and also increases labor requirements.

Japanese Patent Laid-Open No. 1-287083 discloses a method of removing excess water, but this method cannot be commonly used because it requires a special container or a vapor heater.

In addition, in Japanese Patent Application Nos. 6-323225 and 7-5013 the inventors of the present invention have proposed methods in which amorphous siliceous material or silicate material, which reacts well with the calcareous material, is used as part of the siliceous material, or a curing agent such as Portland cement or granulated blast furnace slag is added, but these methods use expensive materials and increase the bulk specific gravity.

Further, the inventors of the present invention have proposed production methods for lightweight calcium silicate boards in which the strength of the compact (green sheet) before hydrothermal reaction and the interlaminar strength thereof is increased, without the addition of siliceous material or silicate material or of curing agents such as Portland cement or granulated blast furnace slag, by using hemihydrate gypsum (Japanese Patent Appln. No. 7-138040) or anhydrous gypsum (Japanese Patent Appln. No. 7-169951) and interlaminar peeling or bursting does not occur during hydrothermal reaction.

However, when hemihydrate gypsum is used as a curing agent hydration occurs extremely quickly if the hemihydrate gypsum is used in slurry form and it becomes necessary to add a setting retarder to the slurry. And when anhydrous gypsum is used as a curing agent it becomes necessary to add a curing accelerator to the slurry, but adding setting retarders and curing accelerators and controlling the amounts added has made the manufacturing process for calcium silicate boards complicated.

SUMMARY OF THE INVENTION

Thus, the objects of the present invention are to provide a method of manufacture for a lightweight calcium silicate board which does not peel or burst during hydrothermal reaction by using alunites, alums, or aluminum sulfate having a specific surface area not requiring the addition of setting retarders or curing accelerators to the curing agent to increase the strength of the compact (green sheet) before hydrothermal reaction and the interlaminar strength thereof, and to provide such lightweight calcium silicate boards.

The present invention provides a method of manufacturing a lightweight calcium silicate board consisting of hydrothermally reacting in a pressure vessel a compact obtained by laminating, using the Hatschek sheet machine process, a raw material slurry containing as its solid content 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers, characterized in that said raw material slurry contains 2 to 20 percent by weight of one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more, and the compact obtained by Hatschek sheet machine process is subjected to primary curing under conditions where

$$(\text{curing temperature} - 15°C) \times \text{curing time} \geqq 120°C \cdot hr \quad (1)$$

and is then hydrothermally reacted.

Further, the present invention provides a method of manufacturing a lightweight calcium silicate board consisting of hydrothermally reacting in a pressure vessel a compact obtained by laminating, using the Hatschek sheet machine process, a raw material slurry containing as its solid content 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers, characterized in that at least one of the slurries in the first and last slurry tanks of a cylinder mesh type Hatschek sheet machine is said raw material slurry with 2 to 20 percent by weight of one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more added to it, and the compact obtained by Hatschek sheet machine process is subjected to primary curing under conditions where

$$(\text{curing temperature} - 15°C) \times \text{curing time} \geqq 120°C \cdot hr \quad (1)$$

and is then hydrothermally reacted.

In addition, the present invention provides a method of manufacturing a lightweight calcium silicate board consisting of hydrothermally reacting in a pressure vessel a compact obtained by laminating, using the Hatschek sheet machine process, a raw material slurry containing as its solid content 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers, characterized in that one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more is or are applied to the extracted film in a powder or slurry form at a rate of 3 to 50 g/m^2 by dry solid content between the making roll and the return roll of the Hatschek sheet machine until the laminate attains a specific thickness, and then the compact obtained by Hatschek sheet machine process is subjected to primary curing under conditions where

$$(\text{curing temperature} - 15°C) \times \text{curing time} \geqq 120°C \cdot hr \quad (1)$$

and is then hydrothermally reacted.

Further, the present invention provides a method of manufacturing a lightweight calcium silicate board consisting of hydrothermally reacting in a pressure vessel a compact obtained by laminating, using the Hatschek sheet machine process, a raw material slurry containing as its solid content 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers, characterized in that the slurry in either the first or the last slurry tank of a cylinder mesh type Hatschek sheet machine process contains more than 20 percent by weight but less than 98 percent by weight of one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more, 2 to 8 percent by weight of fibrous material, and less than 78 percent by weight of one or more species selected from a group consisting of calcareous material and inorganic fillers, and is extracted within the scope of

$$(a)/(b) \times 100 = 1 \text{ to } 10 \quad (2)$$

and

$$(a)/(b) \times 100 \times (c) = 50 \text{ to } 400 \quad (3)$$

wherein (a) is the extracted thickness of said slurry, (b) is the total extracted thickness extracted by the felt of a cylinder mesh type Hatschek sheet machine in one revolution, and (c) is the ratio (in percent by weight) of alunites, alums and aluminum sulfate in said slurry, and the compact obtained by Hatschek sheet machine process is subjected to the primary curing under conditions where

$$(\text{curing temperature} - 15°C) \times \text{curing time} \geqq 120°C \cdot hr \quad (1)$$

and is then hydrothermally reacted.

In addition, the present invention provides a lightweight calcium silicate board (hereinafter simply "calcium silicate board") characterized in that it is a calcium silicate board manufactured according to one of the above manufacturing methods and its interlaminar peeling strength is at least 3 percent of its flexural strength.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is an illustration of one example of a cylinder mesh type Hatschek sheet machine which could be used for the third embodiment of the present invention.

PREFERRED EMBODIMENTS

The basic composition of the raw material slurry used in the method of manufacture of calcium silicate boards according to the present invention is a conventional one containing 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers as its solid content.

Here, examples of the calcareous material that can be used include, for example, slaked lime or quicklime. It is not desirable for the proportion of calcareous material to be less than 17 percent by weight or greater than 50 percent by weight because flexural strength diminishes and dimensional variance increases due to water absorption.

Further, examples of the siliceous material that can be used include, for example, silica sand, diatomaceous earth, or fly ash. It is not desirable for the proportion of siliceous material to be less than 15 percent by weight or greater than 45 percent by weight because flexural strength diminishes and dimensional variance increases due to water absorption.

Further, within the ratios given above for calcareous and siliceous materials 2 to 20 percent by weight of calcareous material and 2 to 25 percent by weight of siliceous material can be used in gel form. An example of gelation conditions would be 75 to 180°C for 1.5 to 4 hours.

In addition, examples of the fibrous material that can be used include, for example, cellulose fiber, polypropylene, vinylon, glass fiber, carbon fiber and the like. It is not desirable for the ratio of fibrous material to be less than 2 percent by weight because flexural strength diminishes, or to be greater than 8 percent by weight because flame resistance is lost. Further, it is desirable to keep the ratio of polypropylene, vinylon, glass fiber, carbon fiber and the like at 5 percent by weight or less if they are used.

Further, examples of the inorganic fillers that can be used include, for example, perlite, wollastonite, mica, talc, calcium carbonate, gypsum and the like. It is not desirable for the ratio of inorganic fillers to be less than 5 percent by weight because dimensional variance increases due to water absorption, or to be greater than 40 percent by weight because flexural strength diminishes.

The method of manufacturing a calcium silicate board according to the present invention is characterized by the following four processes:

According to the method of manufacture as it relates to the first embodiment of the present invention, it is possible to manufacture a calcium silicate board by adding 2 to 20 percent by weight of one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more to a raw material slurry having the previously mentioned ratio of components, and laminating and molding said raw material slurry using the Hatschek sheet machine process, next inducing the calcium originally contained in the calcareous material to react with the alunites, alums, and/or aluminum sulfate by primary curing of the compact thus obtained, and then subjecting the compact to hydrothermal reaction.

In other words, the raw material slurry used in the first embodiment contains as its solid content 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers, and 2 to 20 percent by weight of one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more. Here, it is not desirable for the ratio of alunites, alums, and/or aluminum sulfate to be less than 2 percent by weight because the interlaminar strength of the green sheet is poor, or to be greater than 20 percent by weight because flexural strength diminishes. An addition of 5 to 15 percent by weight is preferable.

In the present invention, alunites with a Blaine specific surface area of 4000 cm^2/g or more means alunites containing potassium and/or sodium. It is not desirable for the Blaine specific surface area of the alunites to be less than 4000 cm^2/g because reactivity with the calcium from the calcareous material is poor, the strength of the green sheet is insufficient, and peeling occurs. Further, alums with a Blaine specific surface area of 4000 cm^2/g or more means alums containing potassium and/or sodium. It is not desirable for the Blaine specific surface area of the alunites to be less than 4000 cm^2/g because reactivity with the calcium from the calcareous material is poor, the strength of the green sheet is insufficient, and peeling occurs. In addition, it is not desirable for the Blaine specific surface area of the alu-

minum sulfate to be less than 2000 cm^2/g because reactivity with the calcium from the calcareous material is poor, the strength of the compact (green sheet) immediately after the Hatschek sheet machine process is insufficient, and peeling occurs.

According to the first embodiment of the present invention, a raw material slurry having the above composition is used to make a compact (green sheet) by the Hatschek sheet machine process. Any conventional process can be substituted for the Hatschek sheet machine process and the invention is not limited thereto.

In the first embodiment of the present invention, the compact obtained in the above manner is not immediately subjected to hydrothermal reaction, it is first cured. Primary curing is carried out under the following conditions:

$$(\text{curing temperature} - 15°C) \times \text{curing time} \geqq 120°C \cdot hr \tag{1}$$

Here, it is not desirable for the primary curing conditions, i.e., the value of equation (1), to be less than 120°C · hr because the compact cannot attain sufficient strength due to inadequate curing. Primary curing requires a curing temperature greater than 15°C and it is preferable to carry out primary curing under conditions of 180°C . hr with the curing temperature in the range of 30 to 80°C.

After the compact has been subjected to primary curing under the aforementioned conditions, it can be made into a calcium silicate board by hydration under conventional hydrothermal conditions and subsequent conventional processing. Hydrothermal reaction can be carried out in a pressure vessel under saturated water vapor pressure at 150 to 200°C, and preferably at 170 to 190°C, for 5 to 20 hours, preferably for 8 to 12 hours.

Next, according to the method of manufacture as it relates to the second embodiment of the present invention, it is possible to manufacture a calcium silicate board by laminating and molding a raw material slurry having the basic composition mentioned above using the Hatschek sheet machine process with at least one of the slurries in the first and last slurry tanks of a cylinder mesh type Hatschek sheet machine being the aforementioned raw material slurry with 2 to 20 percent by weight of one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more added to it, next hydrating the alunites, alums, and/or aluminum sulfate by primary curing of the compact obtained using the Hatschek sheet machine process, and then subjecting the compact to hydrothermal reaction.

In other words, in the second invention a slurry of similar composition to the raw material slurry used in the aforementioned first invention is used as the slurry in the first and/or last slurry tanks of a cylinder mesh type Hatschek sheet machine. Here, it is not desirable for the ratio of alunites, alums, and/or aluminum sulfates in the slurry used in the first and/or last slurry tanks to be less than 2 percent by weight of the solid content because the interlaminar strength of the green sheet manifests itself poorly, or to be greater than 20 percent by weight because flexural strength diminishes.

According to the second invention of the subject invention, a slurry made by adding alunites, alums, and/or aluminum sulfates to a raw material slurry of basic composition is placed in the first and/or last slurry tanks of a cylinder mesh type Hatschek sheet machine and laminated and molded using the Hatschek sheet machine process. The placement of this slurry into the first and/or last slurry tanks of the Hatschek sheet machine is not limited in any way and, for example, a slurry with a different mixture ratio may be made in another box and transferred from a specialized chest, or the alunites, alums, and/or aluminum sulfates can be added to another slurry in a conduit between the chest and the slurry tank.

Next, according to the method of manufacture as it relates to the third embodiment of the present invention, it is possible to manufacture a calcium silicate board by laminating and molding a raw material slurry having the basic composition mentioned above using the Hatschek sheet machine process, applying alunites, alums, and/or aluminum sulfate to the extracted film in a powder or slurry form at a rate of 3 to 50 g/m^2 by dry solid content between the making roll and the return roll of the Hatschek sheet machine until the laminate attains a specific thickness, next hydrating the alunites, alums, and/or aluminum sulfate by primary curing of the compact obtained using the Hatschek sheet machine process, and then subjecting the compact to hydrothermal reaction.

It is not desirable for the distribution of alunites, alums, and/or aluminum sulfates to be less than 3 g/m^2 by dry solid content because the interlaminar strength of the green sheet is poor, or for said distribution to be greater than 50 g/m^2 because a layer of alunites, alums, and/or aluminum sulfates forms, cracks develop on drying, and flexural strength diminishes.

Further, the concentration of dry solid content in the distributing slurry is not limited in any way, but it is preferable for concentration of dry solid content to be slightly higher than the concentration of dry solid content of slurry generally used in the Hatschek sheet machine process, usually 5 to 20 percent by weight but preferably 8 to 15 percent by weight.

An example of a cylinder mesh type Hatschek sheet machine which could be used in the third embodiment of the present invention is shown in Figure 1. According to this cylinder mesh type Hatschek sheet machine, a green sheet is made by transferring a slurry (7) to an extractor felt (3) by a circular mesh cylinder (8) disposed in a slurry tank (6), this operation being repeated as many times as there are slurry tanks (or circular mesh cylinders) to form an extracted

film (4), then winding this extracted film (4) around a making roll (1) a specific number of times until it attains a specific thickness, and cutting it. Because peeling and bursting generally occurs between the successive layers of film superposed on each other on the making roll (1), it is possible to improve the adhesion (interlaminar strength) between said layers of film, and thus prevent pealing and bursting, by disposing an application device (5) between the return roll (2) and the making roll (1) and applying alunites, alums, and/or aluminum sulfate in a powder or slurry form.

Next, according to the method of manufacture as it relates to the fourth embodiment of the present invention, it is possible to manufacture a calcium silicate board by laminating and molding a raw material slurry having the basic composition mentioned above using the Hatschek sheet machine process with the slurry in either the first or the last slurry tank of a cylinder mesh type Hatschek sheet machine containing more than 20 percent by weight but less than 98 percent by weight of alunites, alums and/or aluminum sulfate, 2 to 8 percent by weight of fibrous material, and less than 78 percent by weight of one or more species selected from a group consisting of calcareous material and inorganic fillers, being extracted within the scope of

$$(a)/(b) \times 100 = 1 \text{ to } 10 \qquad (2)$$

and

$$(a)/(b) \times 100 \times (c) = 50 \text{ to } 400 \qquad (3)$$

wherein (a) is the extracted thickness of said slurry, (b) is the total extracted thickness extracted by the felt of a cylinder mesh type Hatschek sheet machine in one revolution, and (c) is the ratio (in percent by weight) of alunites, alums and aluminum sulfate in said slurry, next hydrating the alunites, alums, and/or aluminum sulfate by primary curing of the compact obtained using the Hatschek sheet machine process, and then subjecting the compact to hydrothermal reaction. It is not desirable for the values shown in expression (2) to be less than 1 because adhesion between the layers of the film diminishes, and further, it is not desirable for the values shown in expression (2) to be greater than 10 because there is a tendency for the strength to decrease. Further, it is not desirable for the values shown in expression (3) to be less than 50 because adhesion between the layers of the film diminishes, and further, it is not desirable for the values shown in expression (3) to be greater than 400 because there is a tendency for the strength to decrease.

Calcium silicate boards manufactured according to the methods of manufacture as they relate to the first through fourth inventions of the present invention show extremely superior values for interlaminar peeling strength which is at least 3% of their flexural strength. Flexural strength has been evaluated according to JIS A 5418 using a No. 3 test piece, and tests for interlaminar peeling strength were carried out on a 30 x 30 mm test piece.

As can be seen from the above descriptions, using the method of manufacture for a calcium silicate board according to the present invention, it is possible to improve the overall strength and interlaminar strength of the compact before hydrothermal reaction if the compact is formed from a raw material slurry by the Hatschek sheet machine process, and thus it is possible to prevent peeling and bursting of the compact during hydrothermal reaction as well as to improve the interlaminar peeling strength of the calcium silicate board thus obtained.

EXAMPLES

Example 1

Materials were blended in the ratios shown in Table 1, mixed with 12 times as much water and stirred. The gel was synthesized from 10 percent by weight of slaked lime and 10 percent by weight of diatomaceous earth (weight ratio 1:1) at 90°C for 2 hours. Water was further added to the mixture thus obtained to form a raw material slurry with a solid content of 3 percent by weight, and compacts were extracted to a thickness of 6 mm.

Next, the obtained compacts (green sheets) were subjected to primary curing in a moist atmosphere under the conditions shown in Table 1, then subjected to hydrothermal reaction in saturated water vapor in a pressure vessel at 180°C for 10 hours.

Table 1 shows the bulk specific gravity, flexural strength, and interlaminar peeling strength (all in absolute dry condition) after hydrothermal reaction.

In the subject example, the Blaine specific surface area of Alunite #1 was 10200 cm^2/g, the Blaine specific surface area of Alunite #2 was 3720 cm^2/g, the Blaine specific surface area of Alum #1 was 4260 cm^2/g, the Blaine specific surface area of Alum #2 was 3150 cm^2/g, the Blaine specific surface area of Aluminum Sulfate #1 was 2630 cm^2/g, and the Blaine specific surface area of Aluminum Sulfate #2 was 1840 cm^2/g.

Table 1

	Examples				Comparative Examples						
	1	2	3	4	1	2	3	4	5	6	7
Blending Ratio (wt%):											
Slaked lime	31	25	27	29	32	20	27	29	31	30	31
Silica sand	30	24	27	28	31	19	27	28	30	29	30
Gel	20	20	20	20	20	20	20	20	20	20	20
Wollastonite	10	10	10	10	10	10	10	10	10	10	10
Pulp	5	5	5	5	5	5	5	5	5	5	5
Glass fiber	1	1	1	1	1	1	1	1	1	1	1
Alunite #1	3	--	--	1	1	--	--	1	--	--	--
Alunite #2	--	--	--	--	--	--	--	--	3	--	--
Alum #1	--	15	--	6	--	25	--	6	--	--	--
Alum #2	--	--	--	--	--	--	--	--	--	5	--
Aluminum Sulfate #1	--	--	10	--	--	--	10	--	--	--	--
Aluminum Sulfate #2	--	--	--	--	--	--	--	--	--	--	3
Primary Curing:											
Temp. (°C)	50	30	80	50	50	30	30	50	50	30	80
Time (hrs)	6	8	6	6	6	8	6	3	6	8	6
Value for Equation (1)	210	120	390	210	210	120	90	105	210	120	390
Bulk specific gravity	0.63	0.62	0.63	0.64	0.62	0.63	0.61	0.61	0.62	0.63	0.63
Flexural strength (kg/cm^2) *1	98	93	94	98	82	71	74	78	73	82	78
Interlaminar peeling strength (kg/cm^2) *2	7.1	8.5	9.3	9.1	1.4	8.8	1.1	0.9	1.0	2.2	1.8
*2/*1x100	7.2	9.1	9.9	9.3	1.7	12.4	1.5	1.2	1.4	2.7	2.3
Peeling during hydrothermal reaction	No	No	No	No	Yes	No	Yes	Yes	Yes	Yes	Yes

Example 2

Materials were blended in the ratios shown in Table 2, mixed with 12 times as much water and stirred. The gel was synthesized from 10 percent by weight of slaked lime and 10 percent by weight of diatomaceous earth (weight ratio 1:1) at 90°C for 2 hours. Water was further added to the mixture thus obtained to form a raw material slurry with a solid content of 3 percent by weight. In addition, a different slurry with a solid content of 10 percent by weight prepared as described in Table 2 was used in the first slurry tank (shown as a 1 in Table 2) and/or the last slurry tank (shown as a 4 in Table 2) and compacts were extracted to a thickness of 6 mm.

Next, the obtained compacts (green sheets) were subjected to primary curing in a moist atmosphere under the conditions shown in Table 2, then subjected to hydrothermal reaction in saturated water vapor in a pressure vessel at 180°C for 10 hours.

Table 2 shows the bulk specific gravity, flexural strength, and interlaminar peeling strength (all in absolute dry condition) after hydrothermal reaction.

In the subject example, the Blaine specific surface area of the alunite was 10200 cm^2/g, the Blaine specific surface area of the alum was 4260 cm^2/g, and the Blaine specific surface area of the aluminum Sulfate was 2630 cm^2/g.

Table 2

	Examples				Comparative Examples		
	1	2	3	4	1	2	3
Blending Ratio (wt%):							
Slaked lime	32	32	32	32	32	32	32
Silica sand	31	31	31	31	31	31	32
Gel	20	20	20	20	20	20	20
Wollastonite	10	10	10	10	10	10	10
Pulp	6	6	6	6	6	6	6
Glass fiber	1	1	1	1	1	1	1
Blending Ratio in first and/or last slurry tank (wt%):							
Slurry tank No	1&4	1	4	4	1&4	1	4
Alunite	3	–	–	5	1	–	–
Alum	–	15	–	5	–	25	–
Aluminum Sulfate	–	–	10	–	–	–	10
Slaked lime	31	27	28.5	28.5	31.5	24	28.5
Silica sand	30	26.5	28	28	30.5	23.5	28
Gel	19.5	17	18	18	20	15	18
Wollastonite	9.5	8.5	9	9	10	7.5	9
Pulp	6	5	5.5	5.5	6	4.5	5.5
Glass fiber	1	1	1	1	1	0.5	1
Primary Curing:							
Temp. (°C)	50	30	80	30	50	30	30
Time (hrs)	6	8	6	8	6	8	6
Value for Equation (1)	210	120	390	120	210	120	90
Bulk specific gravity	0.63	0.61	0.61	0.63	0.64	0.61	0.63
Flexural strength (kg/cm^2)*1	102	87	92	96	75	72	81
Interlaminar peeling strength (kg/cm^2)*2	7.2	6.9	6.6	7.1	1.2	9.0	11
*2/*1 x 100	7.1	7.9	7.2	7.4	1.6	12.5	1.4
Peeling during hydrothermal reaction	No	No	No	No	Yes	No	Yes

Example 3

Materials were blended in the ratios shown in Table 3, mixed with 12 times as much water and stirred. The gel was synthesized from 10 percent by weight of slaked lime and 10 percent by weight of diatomaceous earth (weight ratio 1:1) at 90°C for 2 hours. Water was further added to the mixture thus obtained to form a raw material slurry with a solid content of 3 percent by weight, and compacts were extracted to a thickness of 6 mm. During extraction, alunite, alum and aluminum sulfate were applied to the extracted film between the return roll and the making roll in a powder or slurry form in the amounts shown in Table 3. The slurry used had a solid content of 10 percent by weight.

Next, the obtained compacts (green sheets) were subjected to primary curing in a moist atmosphere under the conditions shown in Table 3, then subjected to hydrothermal reaction in saturated water vapor in a pressure vessel at 180°C for 10 hours.

Table 3 shows the bulk specific gravity, flexural strength, and interlaminar peeling strength (all in absolute dry condition) after hydrothermal reaction.

In the subject example, the Blaine specific surface area of the alunite was 10200 cm^2/g, the Blaine specific surface area of the alum was 4260 cm^2/g, and the Blaine specific surface area of the aluminum Sulfate was 2630 cm^2/g.

Table 3

	Examples				Comparative Examples		
	1	2	3	4	1	2	3
Blending Ratio (wt%):							
Slaked lime	32	32	32	32	32	32	32
Silica sand	32	32	32	32	32	32	32
Gel	20	20	20	20	20	20	20
Wollastonite	10	10	10	10	10	10	10
Pulp	5	5	5	5	5	5	5
Glass fiber	1	1	1	1	1	1	1
Application condition:							
(P=powder, S=slurry)	P	S	P	P	P	S	P
Alunite (g/m^2)	3	–	–	10	3	–	–
Alum (g/m^2)	--	10	–	10	–	1	–
Aluminum Sulfate	–	–	40	–	–	–	60
Primary Curing:							
Temp. (°C)	50	50	30	50	30	50	30
Time (hrs)	6	6	8	6	6	6	8
Value for Equation (1)	210	210	120	210	90	210	120
Bulk specific gravity	0.63	0.62	0.64	0.63	0.61	0.62	0.65
Flexural strength (kg/cm^2)*1	97	90	82	86	74	75	70
Interlaminar peeling strength (kg/cm^2)*2	6.9	7.8	8.0	7.1	1.1	0.9	4.2
*2/*1 x 100	7.1	8.7	9.8	8.3	1.5	1.2	6.0
Peeling during hydrothermal reaction	No	No	No	No	Yes	Yes	No
Cracked when dry	No	No	No	No	No	No	Yes

Example 4

Materials were blended in the ratios shown in Table 4, mixed with 12 times as much water and stirred. The gel was synthesized from 10 percent by weight of slaked lime and 10 percent by weight of diatomaceous earth (weight ratio 1:1) at 90°C for 2 hours. Water was further added to the mixture thus obtained to form a raw material slurry with a solid content of 3 percent by weight. In addition, a raw material slurry with a solid content of approx. 2 percent by weight was blended and mixed in the same way and placed in either the first or the last slurry tank, and compacts were extracted to a thickness of 6 mm.

Next, the obtained compacts (green sheets) were subjected to primary curing in a moist atmosphere under the conditions shown in Table 4, then subjected to hydrothermal reaction in saturated water vapor in a pressure vessel at 180°C for 10 hours.

Table 4 shows the bulk specific gravity, flexural strength, and interlaminar peeling strength (all in absolute dry condition) after hydrothermal reaction.

In the subject example, the Blaine specific surface area of the alunite was 10200 cm^2/g, the Blaine specific surface area of the alum was 4260 cm^2/g, and the Blaine specific surface area of the aluminum Sulfate was 2630 cm^2/g.

Table 4

	Examples				Comparative Examples			
	1	2	3	4	1	2	3	4
Blending Ratio (wt%):								
Slaked lime	32	32	32	32	32	32	32	32
Silica sand	32	32	32	32	32	32	32	32
Gel	20	20	20	20	20	20	20	20

Table 4 (continued)

	Examples				Comparative Examples			
	1	2	3	4	1	2	3	4
Blending Ratio (wt%):								
Wollastonite	10	10	10	10	10	10	10	10
Pulp	5	5	5	5	5	5	5	5
Glass fiber	1	1	1	1	1	1	1	1
Blending Ratio in first or fourth slurry tank (wt%):								
Slurry tank No.	4	4	1	4	4	4	1	4
Alunite	–	40	–	35	–	40	–	35
Alum	95	–	–	35	95	–	–	35
Aluminum Sulfate	–	–	22	–	–	–	22	–
Slaked lime	–	53	25	–	–	53	25	–
Calcium carbonate	–	–	50	25	–	–	50	25
Pulp	5	7	3	5	5	7	3	–
Value for equation (2)	3	1.5	8	5	3	0.5	12	6
Ratio of curing agent (%)	95	40	22	70	95	40	22	70
Value for equation (3)	285	60	176	350	285	20	264	420
Primary Curing:								
Temp. (°C)	30	50	50	30	30	50	50	30
Time (hrs)	8	6	6	8	6	6	6	8
Value for Equation (1)	120	210	120	120	90	210	210	120
Bulk specific gravity	0.63	0.63	0.65	0.64	0.62	0.63	0.63	0.64
Flexural strength (kg/cm^2)*1	93	98	87	89	81	72	67	73
Interlaminar peeling strength (kg/cm^2)*2	7.8	7.5	6.1	8.0	1.2	1.0	3.0	5.8
*2/*1 x 100	8.4	7.7	7.0	9.0	1.5	1.4	4.5	7.9
Peeling during hydrothermal reaction	No	No	No	No	Yes	Yes	No	Yes

Claims

1. A method of manufacturing a lightweight calcium silicate board consisting of hydrothermally reacting in a pressure vessel a compact obtained by laminating, using the Hatschek sheet machine process, a raw material slurry containing as its solid content 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers, characterized in that the raw material slurry contains 2 to 20 percent by weight of one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more, and the compact obtained by Hatschek sheet machine process is subjected to primary curing under conditions where

$$(\text{curing temperature} - 15°\text{C}) \times \text{curing time} \geq 120°\text{C} \cdot \text{hr} \qquad (1)$$

and then is hydrothermally reacted.

2. A method of manufacturing a lightweight calcium silicate board consisting of hydrothermally reacting in a pressure vessel a compact obtained by laminating, using the Hatschek sheet machine process, a raw material slurry containing as its solid component 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers, characterized in that at least one of the slurries in the first and last slurry tanks of a cylinder mesh type Hatschek sheet machine is the aforementioned raw material slurry with 2 to 20 percent by weight of one or more species

selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more added to it, and the compact obtained by Hatschek sheet machine process is subjected to primary curing under conditions where

$$(\text{curing temperature} - 15°C) \times \text{curing time} \geqq 120°C \cdot hr \qquad (1)$$

and then is hydrothermally reacted.

3. A method of manufacturing a lightweight calcium silicate board consisting of hydrothermally reacting in a pressure vessel a compact obtained by laminating, using the Hatschek sheet machine process, a raw material slurry containing as its solid content 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers, characterized in that one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more is or are applied to the extracted film in a powder or slurry form at a rate of 3 to 50 g/m^2 by dry solid content between the making roll and the return roll of the Hatschek sheet machine until the laminate attains a specific thickness, and then the compact obtained by Hatschek sheet machine process is subjected to primary curing under conditions where

$$(\text{curing temperature} - 15°C) \times \text{curing time} \geqq 120 \cdot hr \qquad (1)$$

and then is hydrothermally reacted.

4. A method of manufacturing a lightweight calcium silicate board consisting of hydrothermally reacting in a pressure vessel a compact obtained by laminating, using the Hatschek sheet machine process, a raw material slurry containing as its solid content 17 to 50 percent by weight of calcareous material, 15 to 45 percent by weight of siliceous material, 2 to 8 percent by weight of fibrous material, and 5 to 40 percent by weight of inorganic fillers, characterized in that the slurry in either the first or the last slurry tank of a cylinder mesh type Hatschek sheet machine process contains more than 20 percent by weight but less than 98 percent by weight of one or more species selected from alunites and alums with a Blaine specific surface area of 4000 cm^2/g or more or aluminum sulfate with a Blaine specific surface area of 2000 cm^2/g or more, 2 to 8 percent by weight of fibrous material, and less than 78 percent by weight of components comprising one or more species selected from a group consisting of calcareous material and inorganic fillers, and is extracted within the scope of

$$(a)/(b) \times 100 = 1 \text{ to } 10 \qquad (2)$$

and

$$(a)/(b) \times 100 \times (c) = 50 \text{ to } 400 \qquad (3)$$

where (a) is the extracted thickness of said slurry, (b) is the total extracted thickness extracted by the felt of a cylinder mesh type Hatschek sheet machine in one revolution, and (c) is the ratio (in wt%) of alunites, alums and aluminum sulfate in said slurry, and the compact obtained by Hatschek sheet machine process is subjected to primary curing under conditions where

$$(\text{curing temperature} - 15°C) \times \text{curing time} \geqq 120°C \cdot hr \qquad (1)$$

and then is hydrothermally reacted.

5. A lightweight calcium silicate board characterized in that it is a calcium silicate board manufactured according to any one of the manufacturing methods set forth in Claims 1 to 4 and its interlaminar peeling strength is at least 3% of its flexural strength.

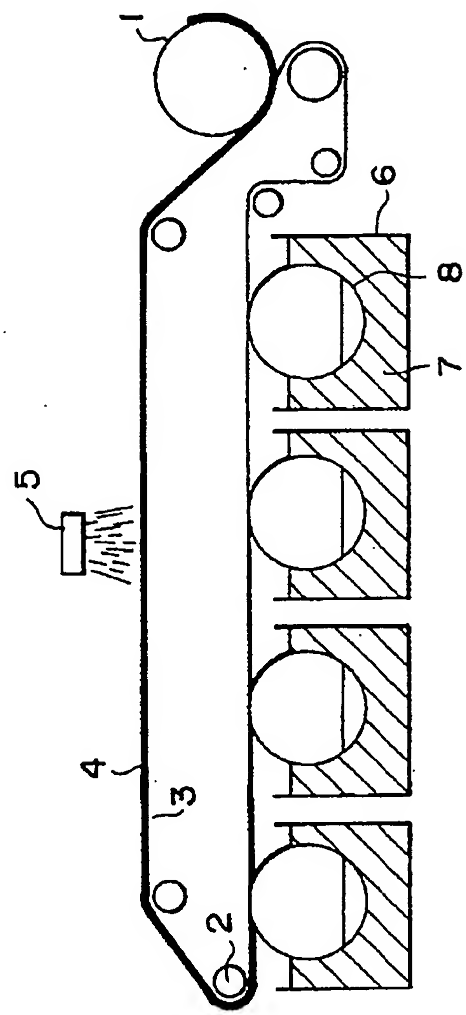


FIG. 1

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 97 30 1638

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
Y,P	PATENT ABSTRACTS OF JAPAN vol. 097, no. 002, 28 February 1997 & JP 08 253375 A (ASK), 1 October 1996, * abstract *	1,2,5	C04B28/18 C04B22/14 C04B40/02 //(C04B28/18, 14:42,18:24, 22:14)
Y	US 3 902 913 A (J.L.HELSER ET AL.) * example 1 *	1,2,5	
A	CHEMICAL ABSTRACTS, vol. 84, no. 20, 17 May 1976 Columbus, Ohio, US; abstract no. 139950n, T. OTOUMA ET AL: page 301; XP000189103 * abstract * & JP 75 151 930 A (ID.)	1-5	
A	DATABASE WPI Week 9304 Derwent Publications Ltd., London, GB; AN 93-030938 XP002036610 & JP 04 357 182 A (ASAHI CHEM IND CO LTD) * abstract *	1-5	TECHNICAL FIELDS SEARCHED (Int.Cl.6) C04B
A	FR 2 243 914 A (THE ASSOCIATED PORTLAND CEMENT MANUFACTURERS LIMITED)		

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	31 July 1997	Daeleman, P

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons
& : member of the same patent family, corresponding document